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(54) Aqueous fluoride-free aluminium cleaning composition

(57) A composition suitable for cleaning aluminium surfaces at moderate to low temperatures contains controlled amounts of a surfactant or combination of surfactants e.g. a mixture of hydrocarbon derivative and abietic acid derivative surfactants, sulphate ions,

phosphate ions and hydrogen ions to provide a pH not in excess of about 2. The aqueous acidic cleaning composition is effective to remove residual organic contaminants as well as metallic particles or smut from the aluminium surfaces achieving a desired degree of surface etching and conditioning the surface for further treatment without the need for fluoride ions.

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SPECIFICATION

Fluoride-free aluminium cleaning composition and process

The present invention broadly relates to the art 5 of cleaning aluminium surfaces and is of particular value in the cleaning of drawn and ironed aluminium container bodies of the types employed in the packaging of foodstuffs and beverages.

In order that the container bodies or cans can 10 be effectively processed through succeeding treating steps to impart satisfactory corrosion protection and lacquer and ink adhesion while at the same time preserving the shiny metallic aluminium appearance of the container, it is necessary to clean such surfaces to remove residual organic lubricants and drawing agents in addition to any metallic particles, commonly referred to as smut, produced during the forming operations. Various acidic and alkaline cleaning 20 compositions have been proposed for this purpose, including those described in U.S. Patent Nos. 3 969 135, 4 009 115 and 4 116 853. Such compositions have generally employed fluoride ions either as essential or preferred ingredients, 25 since they accelerate chemical attack on the aluminium surface, and the removal of metallic particles or smut, and achieve appropriate etching of the surface without impairing the bright shiny aluminium appearance thereof while operating at moderate temperatures, typically 49 to 60°C.

While the effectiveness of fluoride ions to attack and remove the adherent oxide film on aluminium surfaces at moderate cleaning temperatures provides for substantial savings in 35 energy in comparison to cleaners operating at 82°C or higher, the presence of fluoride ions in commercial waste effluents has become environmentally disfavoured over recent years in many areas and there has been a continuing need for providing an aqueous cleaning composition which is fluoride-free while retaining the beneficial cleaning characteristics of such prior art aluminium cleaning compositions. It is also important that such a fluoride-free aluminium cleaning composition can commercially operate at 110 moderate to relatively low temperatures of about 38 to 71°C to provide satisfactory cleaning within commercially acceptable time periods thereby

providing substantial savings in energy costs over cleaners operating at relatively high temperatures of about 82°C and higher. We have now discovered that it is possible to

formulate a fluoride-free aqueous acidic cleaning composition which can be effectively employed at moderate temperatures as low as about 38 to 60°C and which can effectively remove surface contamination and smut from aluminium surfaces within commercially satisfactory time periods to render such surfaces suitable for further treatment in accordance with conventional container manufacturing processing. The composition can be of reasonable cost, can be simple to control, can give effective performance and can overcome environmental problems associated with prior art

65 fluoride-containing cleaning solutions.

An aqueous acidic cleaning composition of the invention contains controlled effective amounts of a surfactant or combination of surfactants, sulphate ions, phosphate ions and hydrogen ions in an amount to provide an acidic pH of less than about 2. More specifically the aqueous acidic cleaning composition contains a surfactant or combination of surfactants in an amount of from about 0.1 up to 30 grams per litre (g/l), a

combination of phosphate ions and sulphate ions in an amount of about 2 to 100 g/l of which about 30 to 80% by weight comprise phosphate ions, and hydrogen ions in an amount to provide a pH not in excess of about 2, and preferably from about 0.5 to 1.2 with a pH of about 0.8 being particularly satisfactory.

The aqueous acidic cleaning composition can readily be prepared and replenished employing a concentrated solution of the active constituents as hereinabove set forth containing the surfactant or surfactants in an amount up to about 200 g/l, a combination of phosphate ions and sulphate ions in an amount up to about 500 g/l and hydrogen ions to provide a pH of less than about 0.

90 Aluminium surfaces are cleaned in the invention by contact with the aqueous acidic cleaning composition for a period of time sufficient to effect desired cleaning and etching of the aluminium surface and the contact of the 95 aluminium surface with the composition is then discontinued.

Since making the present invention we have become aware of the contents of U.S. Patent Application 277 560 of 24 June 1981 and British 100 Patent Application 2 100 757 A. Although the British specification describes concentrates and compositions containing sulphuric acid, phosphoric acid and a surfactant or mixture of surfactants the U.S. application of 24 June 1981 105 requires, as essential features, the presence of a combination of 0.25 to 1 g/l high detergency surfactant and 0.25 to 1 g/l low foaming surfactant. Accordingly all description and claims in that British application and which are not restricted to these particular amounts of particular surfactants are not entitled to the 24 June 1981 priority date and instead have a later priority date than the present application.

The particular type of surfactant or combination 115 of surfactants employed will depend to some extent on the types of organic and inorganic contaminants present on the can surfaces to be cleaned and can be adjusted to provide optimum removal of such soils.

120 The surface active agents which can be satisfactorily employed can be anionic, cationic, or nonionic. Typical examples of such surfactants are: Tergitol Anionic - 08 (Union Carbide Corporation) an anionic surfactant believed to be 125 sodium 2-ethyl hexyl sulphate; Trioton DF-16 (Rohm & Haas Co.) a nonionic surfactant believed to be a modified polyethoxylated straight chain alcohol; Polytergent S-505 LF (Olin Corp.) a nonionic surfactant believed to be a modified

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polyethoxylated straight chain alcohol; Surfonic LF-17 (Jefferson Chemical Co.) a nonionic surfactant believed to be an alkyl polyethoxylated ether; Plurafac RA-30 (BASF Wyandotte Corp.) a nonionic surfactant, believed to be a modified oxyethylated straight chain alcohol; Triton X-102 (Rohm & Hass Co.) a nonionic surfactant believed to be an octyl phenoxy poly ethoxy ethanol; Pluronic L61 (BASF Wyandotte, Inc.) a nonionic surfactant, and believed to be a condensate containing only ethylene oxide and propylene oxide chains; Renex 20 (I.C.I. United Stated, Inc.) a nonionic, polyoxyethylene ester of mixed fatty acids and resin acids.

15 The composition preferably comprises a combination of a hydrocarbon-derivative surfactant and an abietic acid-derivative surfactant, which may be present in substantially equal quantities. Suitable materials are described 20 in U.S. Patent No. 3 969 135.

The hydrocarbon-derivative surfactant preferably employed in the cleaner may be represented by the following general formula:

R(OR'),OH

25 Wherein R is an alkyl or alkylaryl group of 8-22 carbon atoms; R' is a divalent radical selected from ethyl, propyl and combinations thereof and n is an integer from 7 to 22. Commercially available surfactants believed to fall within the above 30

general formula include Triton CF-10 from Rohm & Hass Company; Antarox LF-330, Antarox BL-330 and Igenal CA-630 from GAF Corporation; Trycol LF-1 from Emery Industries, Inc. and Plurafac D-25 from BASF Wyandotte Corporation.

35 These surfactants contain both alkyl and alkylaryl R groups, ethoxy and propoxy R' groups with n values ranging from 8 to 16.

The abietic acid-derivative surfactant may be represented by the general formula:

40 A(R'O),H

wherein R' and n are as defined above and A is the abietic acid radical. The abietic acid-derivative surfactant functions conjointly with the hydrocarbon-derivative surfactant to remove all of the types of organic contaminants which may remain on the surface subsequent to ordinary cold forming operations. The desired concentration ranges are the same as those for the hydrocarbonderivative surfactants. Commercially available surfactants are Surfactant AR 150 supplied by Hercules, Inc. and Pegosperse 700-TO supplied by Glyco Chemicals, Inc. Both of these commercially available surfactants serve as source of an abietic acid ester containing approximately 14 to 16 mols 55 of ethoxylation.

The optimum concentration of the hydrocarbon-derivative surfactants is above 0.1 and below 30 g/l. Very low amounts, for instance below 0.1 g/l, give inadequate cleaning and high amounts, for instance above 30 g/l, may be usable but may lead to less satisfactory stability of the

surfactant in the aqueous acidic cleaner. Generally, however, best results are obtained when the amount is from 0.5 to 2.1 g/l, with 65 values of above 1 g/l, and in particular about 1.3 g/l, generally giving best results. As mentioned above the amount of abetic acid-derivatives surfactant is generally similar to the amount of the hydrocarbon-derivatives surfactant and so preferably falls within the same ranges, with preferred values again in the range 0.5 to 2.1 g/l, especially above 1 g/l and most preferably about $1.3 \, g/l.$

It has been found that variations in the alkylene 75 oxide end groups of either of the above surfactants does not adversely affect their efficacy. The final hydroxy group may be replaced, for example, by a chloride substituent. Alkyl or aryl substitutions may also be made.

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The total amount of phosphate and sulphate in the working composition is generally in the range of about 5 to 35 g/l. Since phosphate must supply between about 30 and 80% of the total of phosphate plus sulphate it follows that the 85 amount of phosphate is generally in the range 0.6 to 80 g/l and preferably 1.6 to 30 g/l and the amount of sulphate is generally in the range 0.4 to 70 g/l and preferably 1.4 to 20 g/l.

The phosphate ions can be introduced in the 90 form of any one of a variety of bath compatible alkali metal and ammonium phosphate salts although phosphoric acid itself constitutes the preferred material.

As in the case of the phosphate ions, the

95 sulphate ions can be introduced in the form of any one of a variety of bath compatible salts including alkali metal and ammonium salts although sulphuric acid itself comprises the preferred material. In either event, the phosphate 100 and sulphate ions are introduced to preferably provide a hydrogen ion concentration to provide an acidity of the operating cleaner composition of a pH not in excess of 2 with a pH of about 0.5 to about 1.2 being preferred and a pH of about 0.8 being particularly satisfactory. The appropriate concentration of hydrogen ions to provide the desired acidity can conveniently be achieved by appropriate additions of phosphoric acid and sulphuric acid which simultaneously introduces 110 the requisite quantity of phosphate and sulphate

ions in the solution.

When a bright shiny aluminium appearance is required, the concentration of the combined phosphate and sulphate ions is preferably 115 maintained at a level below about 17 g/l. When a frosty, etched aluminium surface appearance can be tolerated as in the case of over-all painted or decorated containers, the combined concentration of phosphate and sulphate ions can be increased 120 up to about 100 g/l. Since the efficacy of cleaning and etching of the aluminium surface is related to the time, temperature, and acidity of the cleaning treatment, the concentration of the active constituents can be adjusted for any specific 125 situation within the parameters hereinabove defined to attain the desired cleaning and

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appearance of the aluminium surfaces.

The cleaning composition may also optionally contain any one of a variety of commercially available anti-foaming agents in usual amounts in 5 the event objectionable foaming of the cleaning solution occurs depending upon the particular mode of application.

The composition can initially be formulated as a concentrate which can be diluted with water to provide the desired working solution. As mentioned, the concentrate will contain up to about 200 g/l surfactant, up to about 500 g/l phosphate plus sulphate and will have hydrogen ions in an amount sufficient to provide a pH less 15 than 0. In order to distinguish between wording solutions and concentrates it is convenient to define the concentrates as containing 30 to 200 g/l surfactant and 100 to 500 g/l phosphate plus sulphate. The relative proportions of 20 surfactant and sulphate will be fixed by the relative proportions desired in the final working solution. As mentioned it is often preferred that the amount of sulphate in the working solution should be up to 20 g/l (20% of 100 g/l combined 25 sulphate plus phosphate) and the amount of surfactant is from 0.1 to 30 g/l in the working solution. The amount of surfactant per part by weight sulphate in the working solution and in the concentrate is therefore preferably between 0.1 30 parts surfactant to 20 parts sulphate up to 30 parts surfactant to 20 parts sulphate, i.e. 0.005 to 1.5 parts surfactant per part by weight sulphate.

Aluminium surfaces are cleaned in the invention by contact with the working solution at 35 temperatures generally between 38°C and 71°C with temperatures of 44 to 60°C being preferred for energy conservation considerations. Contact may be by immersion, flooding or, preferably, by spray application. Spray is preferred especially in 40 view of the configuration of the containers being cleaned. The duration of contact will depend upon the magnitude and nature of the contaminants present but is generally from as low as 10 seconds up to 5 minutes or more with contacts of 10 seconds to 2 minutes, for instance 30 seconds to 1 minute, being sufficient for most conditions.

The surfaces may, after contact with the acidic cleaning solution, be subjected to rinse and other treatment solutions in conventional manner, generally by conventional spray application.

Considering now in more detail the overall commercial process, the manufacturing sequence for producing aluminium container bodies or cans conventionally comprises providing an aluminium 55 strip which passes through a cupping press which forms a preliminary cup-shaped disc which is transferred to a draw and ironing press producing an elongated cup-shaped body. During the cupping press and draw and ironing 60 operation, various organic lubricants as well as coolants including water or dilute aqueous emulsions are applied to the surfaces of the part to facilitate the forming operations. The container bodies or cans thereafter are transferred to a 65 trimmer in which the upper edge is trimmed

whereafter the trimmed can passes through a washer containing a plurality of cleaning, rinsing and treatment stages. At the conclusion of the washing and treating cycle, the treated cans are 70 transferred to a dry-off oven and the interior and exterior surfaces of the can are subjected to one or a plurality of lacquering steps and exterior decorative printing steps. Typically, the exterior surface of the can is first provided with a

decorative ink printing of suitable indicia which after drying is followed by a conventional exterior can lacquer coating and a curing of the coating by heating to an elevated temperature such as about 204°C for a period of about 10 minutes in a

80 recirculating air oven. After cooling, the interior can lacquer coating is applied to the interior surfaces of the can and the lacquer comprises any of the types conventionally employed which are selected so as to chemically resist the foodstuff or

85 beverages to be placed within the can in a subsequent filling station. The interior lacquer coating is again cured at an elevated temperature and the can may further optionally be subjected to a second exterior lacquer coating followed by an 90 additional cursing step before transfer to the filling station.

In many instances, the filled cans after sealing are subjected to a pasteurization treatment in order to destroy bacteria. Such pasteurization treatment typically comprises immersing the filled and sealed cans in water heated to about 65 to 71°C for a period of about 30 minutes. Such a pasteurization treatment does not effect the overvarnished side walls of the can but unvamished exterior bottom of the container has in many instances undergone discoloration during pasteurization which is highly objectionable.

In order to assure excellent quality and durability of such decorated, varnished and filled cans during processing, shipment, storage and ultimate end use not only with respect to appearance but also with respect to durability, various tests have been devised for measuring adhesion of the lacquer coating to the can 110 surfaces, resistance to corrosion when subjected to high humidity at elevated temperatures, corrosion resistance of the can surfaces and discoloration of the varnished and unvarnished exterior surfaces under various conditions. It is 115 apparent that the cleaning of the can surfaces to remove the contaminating substances thereon

rendering the surfaces susceptible for further treatment and lacquering comprises an important step in the manufacturing sequence and ensuring 120 that the cleaned can surfaces are receptive to such post treatments. Typically, the trimmed can

body from the trimming operation enters the multiple stage washer in which after pre-washing, the can body is exposed to the cleaner of the 125 present invention which is applied such as by

spray application for a period of about 1 minute at a temperature preferably ranging from about 44°C to about 60°C, whereafter the cleaned can is spray water rinsed at about 49°C for a period of 130 about 15 seconds. Thereafter the cleaned and

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rinsed can body is subjected to a treatment of any of the various types known in the art to improve the surface characteristics thereof and to enhance corrosion resistance and other desirable properties whereafter the treated can is again spray water rinsed at about 49°C followed by a 15 second deionized water spray rinse normally at room temperature (21-27°C) whereafter the can is dried in a hot air recirculating oven usually at about 193 to 204°C for a period of 5 minutes. After cooling, the can thereafter is decorated and lacquered as previously described.

The following are some examples of the invention, Example 1 being comparative. In these examples reference is made to the "TR-4" test procedure. This has been developed to evaluate the resistance to discoloration of a cleaned and treated but unlacquered aluminium surface, also called "Bare Corrosion Test" which simulates 20 exposure of the unpainted exterior bottom of an aluminium can during a commercial Pasteurization process. For this purpose, an aqueous solution is prepared simulating a typical water composition employed in the Pasteurization process containing 82.4 parts per million (ppm) sodium chloride, 220 ppm sodium bicarbonate, 2180 ppm of a water conditioner and the balance deionized water to form one litre. The water conditioning agent employed is a proprietary product supplied by DuBois Chemicals, Inc. under the brand designation DuBois 915 which exhibits a total alkalinity of a 5.8% Na₂O and on analysis contains sodium nitrate, carbonate, triethanolamine and a dodecylphenyl polyethylene glycol. The test procedure employing the aforementioned TRtest solution involves subjecting the treated and unlacquered containers to the solution for a specified time, e.g. 30 minutes while maintained at a specified elevated temperature such as 65°C ± 2.5°C. Following the test, the test specimen is removed, rinsed with water, dried and visually inspected for discoloration. Test specimens are rated from 1 (no staining or discoloration) to 10 (dark gold to grey-black discoloration or extensive non-uniform mottling of the surface). Ratings of 1 to 4 are generally considered commercially acceptable in the

50 EXAMPLE 1

are not acceptable.

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For comparison purposes, a fluoride-free aqueous acidic cleaning solution is prepared containing 7.7 g/l sulphuric acid (100%), about 2 g/l of an abietic acid-derivative surfactant 55 commercially designated as AR-150, about 2 g/l of a hydrocarbon-derivative surfactant commercially available under the designation Trycol LF-1, and the balance water. A total of 19 litres of the cleaning solution is provided at a nominal pH of 60 about 1.2.

aluminium can industry whereas ratings of 5 to 10

Aluminium cans from the can trimming operation having residual organic lubricants and coolants on the surface thereof are cleaned with the cleaner for a period of 1 minute by spray

application at a temperature of about 60°C. The cleaned cans thereafter are subjected to a warm tap water rinse at about 49°C for a period of 15 seconds.

The cleaned and rinsed cans are thereafter 70 subjected to a surface treatment employing an aqueous chromium-free treating solution sold under the designation Bonderite K-780 available from Parker Division of Hooker Chemicals & Plastics Corporation. The treating solution is 75 applied at a pH of 3.7 by spray application for a period of 15 seconds at a temperature of about 49°C.

The treated cans following the treatment are again spray water rinsed for a period of 15 seconds with tap water at 49°C followed by a 15 second deionized water spray rinse whereafter the can is dried in a recirculating hot air oven for a period of 5 minutes at 193°C.

The bottom portion of the can is removed as a 85 test sample and is subject to the TR-4 test in accordance with the procedure previously described. An inspection of the unlacquered exterior bottom surface of the test specimen at the conclusion of the TR-4 test reveals a rating of 90 about 10 which is generally commercially unacceptable. At the completion of the cleaning treatment and prior to the conversion coating treatment, the can was observed to be water break-free with no etching of the surfaces thereof.

95 EXAMPLE 2

A cleaning solution is prepared identical to that described in Example 1 with the exception that the solution contained an additional 4.5 g/l of phosphoric acid (100%) and a trimmed aluminium 100 can was processed in accordance with the same sequence as described in Example 1. At the completion of the cleaning treatment, the surface of the can was observed to be water break-free with a little etching of the surface thereof.

105 Exposure of the bottom of the can to the TR-4 test evidenced a rating of 4.

EXAMPLE 3

A cleaning solution is prepared similar to that of Example 1 with the exception that 9 g/l of phosphoric acid (100%) is added and trimmed aluminium cans are processed in accordance with the same sequence as in Examples 1 and 2. At the completion of the cleaning treatment the surface of the can was water break-free with a desirable 115 degree of etching. The can bottom after subjection to the TR-4 test had a rating of 0 which is commercially acceptable.

EXAMPLE 4

An aqueous cleaning solution was prepared 120 similar to that described in Example 3 except that the phosphoric acid concentration was increased to 13.5 g/l and the temperature of the cleaning solution was reduced from 60°C to 54.5°C. The results obtained at the lower temperature and at the higher phosphate ion concentration were similar to those obtained on the test specimen

processed in accordance with Example 3.

The results of Examples 2 to 4 clearly evidence the unexpected synergistic effect of sulphate and phosphate ions in combination with a surfactant to effect satisfactory cleaning and etching of an aluminium can surface to remove organic soils and metallic smut while at the same time retaining the attractive shiny metallic appearance of the can and without excessive etching. Tests conducted employing a solution similar to that of Example 1 to which 19 g/l of hydrochloric acid (100%) were added produced excessive etching of the surface resulting in a blacked-streaked and frosty undesirable exterior appearance and the TRtest results were commercially unsatisfactory. Similarly, the addition of 10.4 g/l of nitric acid to the cleaner formulation of Example 1 resulted in substantially no etching of the surface and TRtest results which are commercially unsatisfactory.

The concentrate described above can be used either for initial make-up of the operating solution or for maintaining the operating bath at the appropriate concentration.

CLAIMS

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1. An aqueous acidic fluoride-free composition suitable for cleaning aluminium surfaces comprising at least one surfactant present in an amount of about 0.1 to 30 g/l, phosphate ions and sulphate ions in a total amount of about 2 to
 100 g/l of which about 30 to 80% by weight comprise phosphate ions, and hydrogen ions present in an amount to provide an acidic pH not

in excess of about 2.

2. A composition according to claim 1, in which hydrogen ions are present in an amount to provide

a pH of about 0.5 to 1.2.

3. A composition according to claim 1, in which hydrogen ions are present in an amount to provide a pH of about 0.8.

4. A composition according to any preceding claim in which the surfactant is present in an amount of about 0.5 to 10 g/l.

5. The composition according to any preceding claim in which the surfactant comprises a mixture of a hydrocarbon-derivative surfactant and an abietic acid-derivative surfactant.

6. A composition according to claim 5, in which the hydrocarbon-derivative surfactant is a compound of the formula: R(OR')_nOH, wherein R is an alkyl or alkylaryl group of 8—22 carbon atoms; 115 R' is a divalent radical selected from ethyl, propyl

and combinations thereof and n is an integer from 7 to 22.

7. A composition according to claim 5 or claim 6, in which the abietic acid-derivative surfactant is a compound of the formula: A(R'O)_nH wherein R' is ethyl, propyl, or a combination thereof and n is an integer from 7 to 22 and A is the abietic acid radical.

8. A composition according to any of claims 5 to 7, in which the hydrocarbon derivative and abietic acid derivative surfactants are present in substantially equal amounts.

A composition according to any preceding

65 claim, in which the combination of phosphate ions and sulphate ions is present in a total amount of about 5 to 35 g/l.

10. A composition according to any preceding claim, in which the hydrogen ions are introduced70 as phosphoric acid and sulphuric acid.

11. A composition according to any of claims 5 to 8, in which the amount of each of the surfactants is above 1 g/l.

12. A composition according to any preceding claim, in which the surfactant is not a mixture of from 0.25 to 1 g/l of a high detergency surfactant and 0.25 to 1 g/l of a low foaming surfactant.

13. A composition according to any preceding claim, in which the amount of sulphate is from 1.480 to 20 g/l and the amount of phosphate is from 1.6 to 30 g/l.

14. A composition according to claim 1, substantially as herein described with reference to any of the Examples.

15. An aqueous acidic fluoride-free concentrate suitable for dilution with water to form a cleaning composition for aluminium surfaces as defined in claim 1 containing up to about 200 g/l surfactant, up to about 500 g/l of a combination of phosphate ions and sulphate ions in which the phosphate ions comprise about 30 to 80% by weight, and hydrogen ions present in an amount sufficient to provide a pH less than 0.

16. A concentrate according to claim 15,
95 containing from 30 to 200 g/l surfactant and from 100 to 500 g/l of the combination of phosphate and sulphate.

17. A concentrate according to claim 15, or claim 16, in which the surfactant comprises a mixture of a hydrocarbon-derivative surfactant and an abietic acid-derivative surfactant.

18. A concentrate according to claim 17 in which the hydrocrabon-derivative surfactant is a compound of the formula: R(OR')_nOH, wherein R
105 is an alkyl or alkylaryl group of 8—22 carbon atoms; R' is a divalent radical selected from ethyl, propyl and combinations thereof and n is an integer from 7 to 22.

19. A concentrate according to claim 17 or claim 18, in which the abietic acid-derivative surfactant is a compound of the formula: A(R'O)_nH wherein R' is ethylene, propyl, or a combination thereof and n is an integer from 7 to 22 and A is the abietic acid radical.

20. A concentrate according to any of claims 17 to 19, in which the hydrocarbon derivative and abietic acid derivative surfactants are present in substantially equal amounts.

21. A concentrate according to any of claims 15 to 20 in which the amount of phosphate is more than 30 g/l and the amount of sulphate is more than 20 g/l and the amount of surfactant is 0.005 to 1.5 parts by weight per part by weight sulphate.

22. A process for cleaning an aluminium surface comprising contacting the surface with a composition according to any of claims 1 to 10 or 14 for a period of time sufficient to effect desired cleaning and etching of the surface and thereafter

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discontinuing contact of the surface with the composition.

- 23. A process according to claim 22, in which the temperature of the solution is 44 to 60°C.
- 24. A process according to claim 22 or claim 23, in which contact is conducted for from 10 seconds to 2 minutes.
- 25. A process according to any of claims 22 to 24, in which the aluminium surface is the surface
- 10 of a drawn and ironed aluminium can.
 - 26. A process according to any of claims 22 to 25, in which the composition is a composition according to any of claims 11 to 13.
 - 27. A process according to claim 22,
- 15 substantially as herein described with reference to any of the Examples.
 - 28. A cleaned aluminium surface made by a process according to any of claims 22 to 27.

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